UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 7,172,663 B2
DATED : February 6, 2007
INVENTOR(S): HAMPDEN-SMITH et al.

It is certified that an error appears or errors appear in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 3
Line 35, delete "quantitites" and insert therefor --quantities--.

Column 11
Line 46, delete “per-liter” and insert therefor -- per liter--;
Line 48, delete the third occurrence of “of”.

Column 12
Line 59, delete “,” after the word “Loss”.

Column 35
Line 41, delete “the”.

Column 53
Line 64, delete “particuarly” and insert therefor --particularly--.

Column 61
Line 64, delete “titanatei” and insert therefor --titanate,--;
Line 65, delete “shoving” and insert therefor --showing--.

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FORM PTO-1050

droplets per cubic centimeter, still more preferably greater than about 1 x 10^7 droplets per cubic centimeter, and most preferably greater than about 5 x 10^7 droplets per cubic centimeter. That the aerosol generator 106 can produce such a heavily loaded aerosol 108 is particularly surprising considering the high quality of the aerosol 108 with respect to small average droplet size and narrow droplet size distribution. Typically, droplet loading in the aerosol is such that the volumetric ratio of liquid feed 102 to carrier gas 104 in the aerosol 108 is larger than about 0.04 milliliters of liquid feed 102 per liter of carrier gas 104 in the aerosol 108, preferably larger than about 0.083 milliliters of liquid feed 102 per liter of carrier gas 104 in the aerosol 108, more preferably larger than about 0.167 milliliters of liquid feed 102 per liter of carrier gas 104, still more preferably larger than about 0.25 milliliters of liquid feed 102 per liter of carrier gas 104, and most preferably larger than about 0.333 milliliters of liquid feed 102 per liter of carrier gas 104.

This capability of the aerosol generator 106 to produce a heavily loaded aerosol 108 is even more surprising given the high droplet output rate of which the aerosol generator 106 is capable, as discussed more fully below. It will be appreciated that the concentration of liquid feed 102 in the aerosol 108 will depend upon the specific components and attributes of the liquid feed 102 and, particularly, the size of the droplets in the aerosol 108. For example, when the average droplet size is from about 2 microns to about 4 microns, the droplet loading is preferably larger than about 0.15 milliliters of aerosol feed 102 per liter of carrier gas 104, more preferably larger than about 0.2 milliliters of liquid feed 102 per liter of carrier gas 104, even more preferably larger than about 0.2 milliliters of liquid feed 102 per liter of carrier gas 104, and most preferably larger than about 0.3 milliliters of liquid feed 102 per liter of carrier gas 104. When reference is made herein to

average temperature within the desired stream temperature range. That mode of operation, however, is not preferred. Also, it is preferred that, in most cases, the maximum average stream temperature not be attained in the furnace 110 until substantially at the end of the heating zone in the furnace 110. For example, the heating zone will often include a plurality of heating sections that are each independently controllable. The maximum average stream temperature should typically not be attained until the final heating section, and more preferably until substantially at the end of the last heating section. This is important to reduce the potential for thermophoretic losses of material. Also, it is noted that as used herein, residence time refers to the actual time for a material to pass through the relevant process equipment. In the case of the furnace, this includes the effect of increasing velocity with gas expansion due to heating.

Typically, the furnace 110 will be a tube-shaped furnace, so that the aerosol 108 moving into and through the furnace does not encounter sharp edges on which droplets could collect. Loss of droplets to collection at sharp surfaces results in a lower yield of particles 112. More important, however, the accumulation of liquid at sharp edges can result in re-release of undesirably large droplets back into the aerosol 108, which can cause contamination of the particulate product 116 with undesirably large particles. Also, over time, such liquid collection at sharp surfaces can cause fouling of process equipment, impairing process performance.

The furnace 110 may include a heating tube made of any suitable material. The tube material may be a ceramic material, for example, mullite, silica or alumina. Alternatively, the tube may be metallic. Advantages of using a metallic tube are low cost, ability to withstand steep temperature gradients and large thermal shocks, machinability and weldability, and ease of providing a seal between the tube and other process equipment.

scanning electron microscopy (SEM), energy dispersive spectroscopy (EOS), thermogravimetric analysis (TGA) and helium pycnometry (for density). Maximum average stream temperatures in the furnace are varied from 600°C to 1100°C. Air is used as a carrier gas.

All of the particles are dense, spheroidal and include a true composite of the alloy and barium titanate phases. Generally, particle density, as a percentage of theoretical, decreases with increasing furnace temperatures.

Fig. 61 shows an SEM photomicrograph of a composite particle including 20 weight percent barium titanate made at 1000°C.

Fig. 62 shows a TEM photomicrograph of composite particles including 20 weight percent barium titanate, made at a furnace temperature of 1000°C, showing areas indicated by EDS to be rich in the Pd/Ag alloy and areas rich in the barium titanate. Fig. 63 shows a TEM photomicrograph of composite particles including 5 weight percent barium titanate made at a furnace temperature of 1000°C, showing areas indicated by EDS to be rich in the Pd/Ag alloy and rich in the barium titanate.

Example 12

This example demonstrates preparation of multi-phase particles including palladium and titania.

A liquid feed is prepared including titanium tetraisopropoxide dissolved in a 2.2 weight percent aqueous solution of palladium nitrate. The liquid feed is converted to an aerosol in a single transducer ultrasonic generator at 1.6 MHz using nitrogen as a carrier gas. The aerosol is converted to particles in a furnace at a temperature of 1100°C. Particles are collected and cooled. The amount of titanium tetraisopropoxide in the liquid feed is varied to produce from about 25 weight percent to about 45 weight percent of titanium oxide in the final particles.